# Validation Test Results from the Vehicle Cabin Atmosphere Monitor

M. R. Darrach<sup>1</sup>, A. Chutjian<sup>2</sup>, B. J. Bornstein<sup>3</sup>, A. P. Croonquist<sup>4</sup>, V. Garkanian<sup>5</sup>, V. R. Haemmerle<sup>6</sup>, W. M. Heinrichs<sup>7</sup>, D. Karmon<sup>8</sup>, J. Kenny<sup>9</sup>, R. D. Kidd<sup>10</sup>, S. Lee<sup>11</sup>, J. A. MacAskill<sup>12</sup>, S. M. Madzunkov<sup>13</sup>, L. Mandrake<sup>14</sup>, T.M. Rust<sup>15</sup>, R. T. Schaefer<sup>16</sup>, J. L. Thomas<sup>17</sup>, and N. Toomarian<sup>18</sup>

Jet Propulsion Laboratory, California Institute of Technology, Pasadena CA 91109

We report on the results from validation testing the Vehicle Cabin Atmosphere Monitor (VCAM). VCAM is an autonomous environmental monitor based on a miniature gas chromatograph/mass spectrometer and is scheduled to be flown to the International Space Station (ISS) on shuttle mission STS-131. VCAM is capable of providing measurements of both ppb levels of volatile constituents and the atmospheric major constituents (nitrogen, oxygen, and carbon dioxide) in a space vehicle or station. It is designed to operate autonomously and maintenance free, approximately once per day, with a self-contained gas supply sufficient for a one-year lifetime. VCAM performance is sufficient to detect and identify 90% of the target compounds specified at the 24-hour Spacecraft Maximum Allowable Concentration level. This paper presents a summary of the pre-flight validation test results for VCAM.

### **Nomenclature**

= Atomic and Molecular Collisions Group

= Development Unit

= Fourier Transform Infrared

AMC

FTIR

DU

GC/DMS = Gas Chromatograph/Differential Mobility Spectrometer GC/MS = Gas Chromatograph/Mass Spectrometer HOSC = Huntsville Operations Support Center = International Space Station ISS = Jet Propulsion Laboratory JPL= Johnson Space Flight Center JSC LS = Laboratory Standard <sup>1</sup> Senior Technologist, Atomic and Molecular Collisions Group, JPL CalTech, Pasadena, CA 91109 <sup>2</sup> Senior Scientist, Atomic and Molecular Collisions Group, JPL CalTech, Pasadena, CA 91109 <sup>3</sup> Scientific Applications Engineer, Instrument Autonomy Group, JPL CalTech, Pasadena, CA 91109 <sup>4</sup> Systems Engineer, Micro-Devices Group, JPL CalTech, Pasadena, CA 91109 <sup>5</sup> Mechanical Engineer, Optical Communications Group, JPL CalTech, Pasadena, CA 91109 <sup>6</sup> Software Engineer, Processing Algorithms and Calibration Engineering, JPL CalTech, Pasadena, CA 91109 <sup>7</sup> System Engineer, Inst System Engineering, JPL CalTech, Pasadena, CA 91109 <sup>8</sup> Program Project Manager, Instruments & Science Data Systems, JPL CalTech, Pasadena, CA 91109 <sup>9</sup> Engineer, Instrument Integration and Test, JPL CalTech, Pasadena, CA 91109 <sup>10</sup> Research Scientist, Planetary Chemistry and Astrobiology, JPL CalTech, Pasadena, CA 91109 <sup>11</sup> Technologist, High Capacity Computing and Modeling, JPL CalTech, Pasadena, CA 91109 <sup>12</sup> Research Scientist, AMC, JPL CalTech, Pasadena, CA 91109 <sup>13</sup> Research Scientist, AMC, JPL CalTech, Pasadena, CA 91109 <sup>14</sup> Informatics Researcher, Machine Learning and Instrument Autonomy Group, JPL CalTech, Pasadena, CA 91109 <sup>15</sup> Propulsion Engineer, SpaceX, 1 Rocket Rd, Hawthorne, CA 90250 <sup>16</sup> Electrical Engineer, Advanced Computer Systems and Technology, JPL CalTech, Pasadena, CA 91109

<sup>18</sup> Program Manager, Advanced Instrument Concepts, JPL CalTech, Pasadena, CA 91109

LDHF = Long Duration Human Spaceflight
 MCA = Major Constituents Analysis
 MSFC = Marshal Space Flight Center
 MCE = Monitor and Control Electronics
 MPLM = Multi-Purpose Logistic Module
 NCO = Numerically-Controlled Oscillator

*NIST* = National Institute of Science and Technology

*ORU* = Orbital Replacement Unit

PC = Preconcentrator
PFU = Protoflight Unit
PE = Processor Electronics
rf = Radiofrequency

SMAC = Spacecraft Maximum Allowable Concentration

STDO = Station Detailed Test Objective

TG = Trace Gas

TReK = Telescience Research Kit

*VCAM* = Vehicle Cabin Atmosphere Monitor

VOA = Volatile Organic Analyzer VOCs = Volatile Organic Compounds

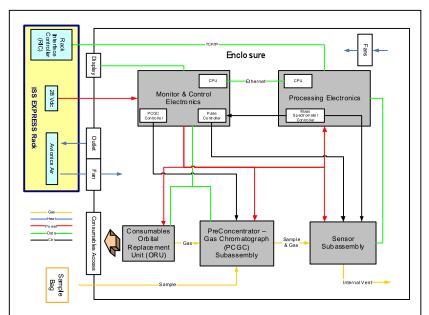
#### I. Introduction

he harmful effects of chemical exposure during long duration human space flight (LDHF) can pose severe health risks to astronauts. Mitigating these effects requires a sensitive monitoring instrument as part of a spacecraft life-support system. Near real-time monitoring of trace volatile organic compounds (VOCs) and the major constituents of the habitat environment is critical to safeguarding astronaut health. Until recently the Volatile Organic Analyzer (VOA)<sup>1, 2</sup> has been the only in-situ analysis tool resident aboard the International Space Station to perform this task. The VOA however has far exceeded its design life and after almost eight years has unfortunately finally ceased operation. To construct additional VOAs as replacements has not been possible because of its relatively large size, complexity, and maintenance difficulties. Other technologies<sup>3, 4</sup> are being flown aboard ISS as Station Detailed Test Objectives (STDOs) to assess their efficacy as possible VOA replacements but satisfying the required specifications remains a very difficult task. A successful environmental monitor must operate autonomously, providing accurate and precise results in the complex ISS cabin environment while satisfying all requirements for sensitivity, identification (of both known and unexpected chemical targets), dynamic range, and instrument mass-volume-power. Examination of the chemicals on the Spacecraft Maximum Allowable Concentration (SMAC) target list illustrates the analytical difficulty of the task. Given the variety and concentrations of these chemicals, coupled with the potential for unexpected and unknown chemical releases into the LDHF environment, a gas chromatograph/mass spectrometer (GC/MS) appears to be the best instrument to address these requirements. It is the standard instrument for analysis of chemicals in terrestrial and planetary environments. GC/MSs have successfully flown on unmanned planetary missions such as Pioneer Venus, Galileo, and Cassini, with comparable instruments to be proposed to Venus, Saturn and Titan. A description of the VCAM GC/MS approach was presented earlier.<sup>5-7</sup> New results are presented summarizing VCAM analytical performance for pre-flight validation. Included are the VCAM concept of operations, and test results for identification and quantification of the complex gas mixtures expected aboard the ISS.

## II. Description of the VCAM

Shown in Fig. 1 is a schematic diagram of the VCAM layout. The air is typically sampled at the VCAM location but can also perform analysis of other locations within the ISS by analyzing samples that have been collected at another location through use a sample bag. For the analysis of cabin-air for VOCs, VCAM operates in TG mode where air is sampled through a filtered inlet and adsorbed onto a PC module. After adsorption of the VOCs onto the PC bed the residual air is purged and VOCs are thermally desorbed in a low flow of helium that is directed through the GC microinjector. At the peak of the chemical thermal-desorption profile the microinjector captures approximately  $20~\mu\ell$  of the stream into the sample loop. This portion is compressed by the pressure of the GC carrier gas, and is injected onto the head of the GC column. The GC elution stream is directed into the center of a Paul ion-trap mass spectrometer. There, a pulsed beam of electrons ionizes the analytes. The resultant ions are

then mass-analyzed by the Paul trap in its so-called selective massinstability mode: the RF amplitude is swept linearly in time, and the ionized species are "walked" off the edge of the Paul trap stability region. The mass/charge-selected ions are ejected onto the front cone of a channel-type electron multiplier, and the mass spectrum stored. The Paul trap electrodes are coated with an inert silanizing layer. Together with an internal halogen bulb which maintains the mass spectrometer at 100°C approximately during operation this ensures surface cleanliness. The PCGC. microinjector, valves, heaters, sample pump, and Paul trap sequencing is controlled by the Monitor and onboard Control Electronics (MCE) and Processor Electronics (PE). The mass spectra are analyzed either autonomously onboard, or the data transmitted to



**Figure 1. Schematic Representation of the VCAM Subassemblies.** The Paul ion trap is contained in the vacuum Sensor Subassembly Module; the calibration and He carrier gases are part of the Consumables ORU.

ground. In addition to the TG Mode, VCAM has a separate operating cycle called the MCA mode configured for the PCGC/MS. Here, cabin air is introduced directly into the microinjector and subsequent GC column, bypassing the PC. In this mode three of the major cabin-air constituents  $(N_2, O_2, \text{ and } CO_2)$  are identified and monitored. This provides dissimilar redundancy to the Major Constituents Analyzer already aboard ISS.

A photograph of the VCAM PFU is shown in Fig. 2. Its mass is 25.2 kg (without consumables) and consumes 140 W (peak) and 100W (nominal) power as derived from the EXPRESS 28V rack. Gas consumables sufficient for one year of operational life are packaged into a separate orbital replacement unit (ORU). Its mass is 5.1 kg. The

consumable gases are contained in two tanks: one of pure helium used as the GC carrier gas, and the other a calibrant gas mixture. The calibration gases that will be used to validate and verify the following key operating parameters of VCAM while on-orbit: (1) the enhancement factor of the preconcentrator, (2) proper operation of the GC column by measuring the elution time and system sensitivity of the calibrant mixture, and (3) the mass range, mass resolution, mass cross talk, and system sensitivity of the Paul trap. Use will be made of a modified TO15 EPA Protocol for testing the instrument stability and response. The TO15 protocol employs daily tests using fluorobenzene (FB, C6H5F, mass 96 amu) and bromofluorobenzene (BFB, C6H4FBr, mass 174 amu for the 79Br isotope and 176 amu for the equally abundant 81Br isotope acetone (12C3H6O, 58 amu). For VCAM only FB and



Figure 2. Photograph of the VCAM Development Unit Protoflight Unit (PFU). Not shown in this picture is the Orbital Replacement Unit (ORU) that contains the helium GC carrier gas and calibrant gas.

acetone (12C3H6O) will be used to calibrate the PCGC at the high- and low-mass ranges of the MS, respectively. VCAM ground-based testing suggests that once/week runs of the calibration gas mixture will be sufficient while on- orbit. Use of these calibrant gases in the closed ISS environment does not pose an astronaut health risk, even in the

event of a total release into the closed ISS. Cooling is by means of forced air supplied from the ISS Avionics air-cooling loop; circulation through the VCAM interior by a pair of internal fans. Note that the VCAM sub-assemblies and packaging have not been optimized for volume as they occupy the standard 64.4 liter EXPRESS rack module. Downlink data communication is through the ISS medium-rate data link, buffered onto the ISS high-rate outage recorder and telemetered to Earth. The data are routed through the White Sands and Huntsville Operations Support Center (HOSC), and then through the internet to JPL where they are presented via the Telescience Research Kit (TReK). Uplink for on-orbit commanding is along the inverse path.

For laboratory science performance testing two additional VCAM assemblies were employed, the Development Unit (DU) and a separate PCGC/MS called the Laboratory Standard (LS). The LS unit has flight form and function, but is packaged in an open architecture. On the DU, LS, and PFU units an extensive series of tests were carried out, consisting of establishing, for each target SMAC species a library of GC elution times and a MS fractionation pattern library which was similar to that of the NIST library. The libraries that fly with VCAM have been validated to provide accurate identifications and quantification for that instrument. Sample bags each containing mixtures ("cocktails") of 5-10 species at concentrations over the required SMAC limit including variations in relative humidity were prepared and analyzed by the VCAM LS, DU, and PFU. Also, canisters containing mixtures of SMAC target species, as well as chemicals not on the SMAC list, were supplied to JPL by the JSC analytical laboratory. During validation testing the autonomous identifications and quantifications were also compared to human interpretations. Following successful completion of the Validation in August 2009, the PFU was delivered to Kennedy Space Center in September 2009 and packed into the Leonardo MPLM in February 2010. Launch aboard STS-131 to the ISS is scheduled for April 5, 2010.

Future developments for VCAM are directed along two paths. The first is to include water-quality monitoring by addition of a water-extraction subassembly that takes advantage of VCAM's modular design. Samples of the habitat's potable water stream are passed over a carboxen preconcentrator bed and the dissolved VOCs are extracted. After several cycles of removing the excess water in a flow of dry He, one heats the PC bed and carries the VOCs in a stream of He onto the microinjector, and thence onto the GC column and into the MS. This subassembly has been tested in the laboratory. It is awaiting integration into VCAM for future use. The second path is to continue the development towards subassemblies having yet lower mass, volume and power. Electronics developments include miniaturization of conventional power supplies to chip size; miniaturization of the RF NCO electronics card to chip size; and use of carbon nanotube arrays to effect ionization of the analytes within the trap.

## III. VCAM Ground Validation

For trace VOC analysis aboard the ISS the targeted SMAC species are divided into three priority classes: Priority 1 species (nine total) including ethanol, acetone, dichloromethane, and perfluoropropane; Priority 2 (16 total) including benzene, C5-C8 alkanes and C3-C8 aldehydes; and Priority 3 (12 total) including 2-butanone, Freon-11, and freon-12. Summarized in Table 1 are the 32 chemical species and their associated concentration ranges over which VCAM was required to autonomously operate. During validation testing on the VCAM units two types of cocktails containing these species were utilized; MK and MU mixes of known and unknown composition, respectively.

For cocktails of known composition, a total of seven MK mixes were developed that each contained groupings of 5-7 of the 32 chemicals. These groupings were chosen to avoid coelutions, based on earlier testing which determined GC elution times. Each of the MK mixes was also prepared at four different dilutions that spanning the required concentration range of the SMAC compounds. An example of two of the MK mixes; MK3 and MK4 are of a single MK dilution series were created and analyzed by both the DU and PFU within a single 24-hr period. The DU analysis of the cocktails served as an independent check of the mix preparation and handling operations. With the exception of two MK5 cocktails, which were supplied by the JSC Toxicology Group, all of the MK cocktails were manufactured in-house by a single person at JPL. Consistent use of the same individual for sample preparation helped to minimize any systematic errors. Sample preparation proceeded as follows: First, a Tedlar (\*Manufacture PN.) was filled with 1 liter of a simulated ISS cabin air atmospheric mix (78.99% N2, 20% O2, 1.01% CO<sub>2</sub>) and hydrated to 50% RH by a syringe injection of distilled de-ionized water. Second, ul quantities of analytic-grade quality pure VOCs were injected through a septum into this sample bag to create a "stock chemical" bag. Third, quantities of the stock vapor were injected into metalized polyethylene sample bags (CalE-Bond. Part No) and using the method of standard dilutions the four required MK cocktails were created. At the completion of MK testing, instrument response curves for each chemical were derived based upon extraction of the mass spectral

ion intensities for each of chemicals in the GC elution peaks. These response curves were then uploaded into the PFU in preparation for quantization verification testing.

For validation of the identification requirements the testing was to be deemed successful if with all results from MK and MU cocktails, taken as an aggregate, the system had identified all of the Priority 1, 2 and 3 compounds 90% of the time. Summarized in Tables 4 – 6 are the VCAM autonomous identification results for the MK cocktails, arranged according to chemical priority. For the MU cocktails, the PFU was 100% successful in identifying all compounds that appeared within its concentration specification range. Across both MK and MU mixes then, the autonomous identification rates exceeded the required 90% value in the requirements. Closer examination of the MK results yield some interesting outcomes. In Priority 1 all chemicals were autonomously identified more than 90% of the time, with the exception of propylene glycol (PG) and octamethylcyclotetrasiloxane (OMCTS). It is recognized within the scientific community that the creation of specific vapor concentrations of certain low-vapor pressure, high boiling point chemicals, such as PG and OMCTS in sample containers is extremely problematic. As such, it was probable that during MK testing the OMCTS and PG concentrations were present at less than the theoretical targeted concentration. In the case of PG, manual examination of the elution data by the science team was not able to detect the chemical in any of the data sets where autonomous identification failed. As such, it is likely that PG was not in these cocktails at all. In the case of OMCTS, manual examination of the elution data by the science team was able to discern the weak presence of OMCTS in some cases where the autonomous identification failed. Further, the extracted OMCTS mass spectral intensities plotted against theoretical targeted concentration revealed a large scatter in the data, confirming that the sample preparation methodology employed for OMCTS was deficient. In Priority 2. all chemicals with the exception of pentanal were detected at rates exceeding 90%. The same sample preparation difficulties as with OMCTS and PG were likely repeated with pentanal since it has a boiling point of 100°C and is difficult to completely volatilize during preparation a sample bag. In Priority 3, all chemicals with the exception of carbonyl sulfide and 1,2-dichloroethane, were autonomously identified at rates exceeding 90%. For 1,2-dichloroethane, one of the two data sets that failed autonomous identification was with a cocktail that was at a concentration below the required range. These 1,2-dichloroethane concentrations were 0.008 ppm and 0.01 ppm, for actual and required concentrations, respectively. In the case of carbonyl sulfide (CS), the chemical co-elutes with air and the air peak comprised of nitrogen, oxygen, and carbon dioxide, is very strong and has intense mass spectral lines which masked the mass spectrum of CS making it extremely difficult to detect autonomously.

| Priority 1<br>Compounds  | Required<br>Concentration<br>Range (ppm) |
|--------------------------|--|
| ethanol                  | 1 – 10                                   |
| acetaldehyde             | 0.1 - 3                                  |
| acetone                  | 0.5 - 5                                  |
| dichloromethane          | 0.03 - 5                                 |
| OMCTS                    | 0.05 - 1                                 |
| HMCTS                    | 0.1 - 2                                  |
| propylene glycol         | 0.5 - 4                                  |
| perfluoropropane         | 10 – 100                                 |
| Priority 2               |  |
| Compounds                |  |
| 1-butanol                | 0.5 -5                                   |
| benzene                  | 0.01 - 1                                 |
| pentane                  | 2 – 20                                   |
| hexane                   | 2 – 20                                   |
| pentanal                 | 0.1 – 2                                  |
| hexanal                  | 0.1 – 2                                  |
| ethyl benzene            | 1 – 10                                   |
| ethyl acetate            | 1 – 10                                   |
| 2-propanol               | 1 – 10                                   |
| Freon 113                | 2 – 10                                   |
| furan                    | 0.01 – 1                                 |
| toluene                  | 1 – 10                                   |
| xylenes (o, m, p)        | 1 – 10                                   |
| Priority 3               |  |
| Compounds                |  |
| 1,2-dichloroethane       | 0.01 - 0.1                               |
| 2-butanone               | 0.5 - 5                                  |
| 4-methyl 2-<br>pentanone | 2 – 10                                   |
| carbonyl sulfide         | 0.01 – 1                                 |
| chloroform               | 0.02 - 1                                 |
| Freon 11                 | 2 – 10                                   |
| isoprene                 | 0.05 - 1                                 |
| limonene                 | 1 – 10                                   |
| vinyl chloride           | 0.05 - 1                                 |

Table 1. Required Species and Concentration Ranges for the VCAM Validation Testing.

| Сонфоинд               | Concentration<br>Range<br>Requirement<br>(ppm) | Test<br>Concentration<br>#MK3-1 | Test<br>Concentration<br>#MK3-2 | Test<br>Concentration<br>#MK3-3 | Test<br>Concentration<br>#MK3-4 |
|------------------------|--|---------------------------------|---------------------------------|---------------------------------|---------------------------------|
| isoprene               | 0.05 - 1                                       | 0.05                            | 0.096                           | 0.2                             | 2.4                             |
| acetone                | 0.5 - 5  | 0.17                            | 0.3                             | 0.8                             | 8.3                             |
| dichloromethane        | 0.03 - 5                                       | 0.06                            | 0.1                             | 0.3                             | 2.9                             |
| C5-Aldehyde (pentanal) | 0.1 - 2  | 0.08                            | 0.2                             | 0.4                             | 4.2                             |
| C6 Aldehyde (hexanal)  | 0.1 - 2  | 0.09                            | 0.2                             | 0.4                             | 4.3                             |
| chloroform             | 0.02 - 1                                       | 0.02                            | 0.0                             | 0.1                             | 1.1                             |

Table 2. Constituents and Concentrations of the MK3 Cocktails Used for the Four-Point Calibration of the VCAM PFU.

| Сонфоили             | Concentration<br>Range<br>Requirement<br>(ppm) | Test<br>Concentration<br>#MK4-1 | Test<br>Concentration<br>#MK4-2 | Test<br>Concentration<br>#MK4-3 | Test<br>Concentration<br>#MK4-4 |
|----------------------|--|---------------------------------|---------------------------------|---------------------------------|---------------------------------|
| furan                | 0.01 -1  | 0.03                            | 0.068                           | 0.2                             | 0.9                             |
| 2-butanone           | 0.5 - 5  | 0.28                            | 0.6                             | 1.4                             | 7.0                             |
| benzene              | 0.01 -1  | 0.04                            | 0.1                             | 0.2                             | 0.9                             |
| 1,2-dichloroethane   | 0.01 -0.1                                      | 0.02                            | 0.0                             | 0.1                             | 0.6                             |
| 1,2-propylene glycol | 0.5 - 4  | 0.23                            | 0.5                             | 1.2                             | 5.9                             |

Table 3. Constituents and Concentrations of the MK4 Cocktails Used for the Four-Point Calibration of the VCAM PFU.

| Priority 1<br>Compounds | ID (%) |
|-------------------------|--------|
| ethanol                 | 100    |
| acetaldehyde            | 100    |
| acetone                 | 100    |
| dichloromethane         | 100    |
| OMCTS                   | 8      |
| HMCTS                   | 92     |
| propylene glycol        | 33     |
| perfluoropropane        | 100    |

Table 4. Autonomous Identification Rates for the Priority 1 Chemicals During MK Series Testing. The low identification rates for PG and OMCTS were due to sample preparation deficiencies.

| Priority 2        | ID (%)  |
|-------------------|---------|
| Compounds         | 10 (70) |
| 1-butanol         | 100     |
| benzene           | 92      |
| pentane           | 100     |
| hexane            | 100     |
| pentanal          | 83      |
| hexanal           | 92      |
| ethyl benzene     | 100     |
| ethyl acetate     | 100     |
| 2-propanol        | 92      |
| freon 113         | 100     |
| furan             | 92      |
| toluene           | 100     |
| xylenes (o, m, p) | 100     |

Table 5. Autonomous Identification Rates for the Priority 2 Chemicals During MK Series Testing.

| ID<br>(%) |
|-----------|
| 83        |
| 100       |
| 100       |
| 18        |
| 100       |
| 100       |
| 100       |
| 100       |
| 100       |
|           |

Table 6. Autonomous Identification Rates for the Priority 3 Chemicals During MK Series Testing. The carbonyl sulfide identification rate was low due to insufficient GC separation from the air peak..

For validation of the accuracy and precision requirements, three MU mixes were supplied by the JSC Toxicology Group. These challenge mixtures had constituents and concentrations that were not known to JPL personnel prior to analysis by the PFU. In order to account for any losses due to storage effects, upon completion of the PFU testing

the residual MU mixes were returned for testing with the JSC Toxicology GC/MS. Mixture MU1 was agreed to be a cocktail of up to eight compounds randomly selected from the requirements list, within ISS simulated air at 50% RH and without coelutants, where ISS simulated air has slightly elevated levels of carbon dioxide and methane. Mixture MU2 was a cocktail of up to ten compounds randomly selected from the requirements list, within ISS simulated air at 50% RH. Mixture MU3 was a cocktail of up to twenty compounds randomly selected from the requirements list, within ISS simulated air at 50% RH. In addition to compounds from the requirement list, the MU3 mixture could also contain species not on the list but that are nevertheless found on-board ISS based on the experience of the JSC Toxicology Group. The concentrations of these species were at the levels normally found on ISS. The PFU accuracy validation was to be deemed successful if the average derived concentration (in mg/m3, or ppm) was within  $\pm 40\%$  of the known amount for all MU testing. The average derived concentration was the average of three runs of each compound in the mixture. Precision validation was successful if three runs of the MU2 Cocktail were completed and the percent Relative Standard Deviation (RSD) for the derived concentration of each compound in the mixture was  $\pm 20\%$  or less.

Shown in Tables 7-9 are the autonomous results of the PFU validation testing of the MU cocktails. In summary, the quantification error in the MU challenge mixtures averaged  $\pm 49\%$  or  $\pm 43\%$  when compared to concentrations based on retest at JSC or the original theoretical target concentrations, respectively. It was concluded by the validation review panel that VCAM successfully met the quantification criterion within reasonable uncertainty while operating in automated mode. Quantification errors caused by sample introduction into the VCAM may contribute to error magnitude and minimizing dead spaces and leakage paths during sample introduction were recommended in follow on work. The results in Tables 7-9 also demonstrate that VCAM successfully met the precision requirement. For 3 runs of the MU2 unknown challenge mixture VCAM had a %RSD =  $\pm 7\%$ , for concentrations within the VCAM development specification range. Further across all MU mixes, VCAM had a %RSD =  $\pm 18\%$ , including those species outside the required concentration range.

| Compound         | Requirement<br>Range<br>(ppm) | JSC<br>Theory<br>(ppm) | JSC<br>Measured<br>(ppm) | JPL<br>Measured<br>Mean<br>(ppm) | JPL<br>% RSD | % Diff<br>from JSC<br>Measurement | % Diff from<br>JSC Theory |
|------------------|-------------------------------|------------------------|--------------------------|----------------------------------|--------------|-----------------------------------|---------------------------|
| perfluoropropane | 10 - 100                      | 40                     | 72                       | 32                               | 12           | -55                               | -19                       |
| isoprene         | 0.05 - 1                      | 0.1                    | 0.08                     | 0.13                             | 15           | 63                                | 31                        |
| acetaldehyde     | 0.1 - 3                       | 1.2                    | 1.3                      | 0.83                             | 10           | -36                               | -31                       |
| dichloromethane  | 0.03 - 5                      | 1                      | 1.65                     | 0.72                             | 6            | -56                               | -28                       |
| ethanol          | 1 - 10                        | 1                      | 0.39                     | 0.05                             | 18           | -88                               | -95                       |
| pentanal         | 0.1 - 2                       | 2                      | 1.33                     | 2.06                             | 31           | 55                                | 3                         |
| toluene          | 1 - 10                        | 1                      | 0.84                     | 0.33                             | 38           | -60                               | -67                       |
| xylene           | 1 – 10                        | 1                      | 0.77                     | 1.38                             | 28           | 79                                | 38                        |

Table 7. Autonomous Results for Validation Testing Using the MU1 Challenge Cocktail.

| Compound     | JPL<br>Requirement<br>Range (ppm) | JSC Mean<br>(ppm) | JPL Mean<br>(ppm) | % RSD | % Diff<br>Between<br>Means |
|--------------|-----------------------------------|-------------------|-------------------|-------|----------------------------|
| benzene      | 0.01 - 1                          | 0.03              | 0.15              | 8     | 380                        |
| l-butanol    | 0.5 – 5                           | 0.08              | 0.03              | 10    | 165                        |
| isoprene     | 0.05 - 1                          | 0.018             | 0.01              | 16    | 28                         |
| acetone      | 0.5 - 5                           | 0.65              | 0.72              | 5     | 5                          |
| 2-propanol   | 1 – 10                            | 0.285             | 0.11              | 8     | 18                         |
| (o-)xylene   | 1 – 10                            | 0.13              | 0.41              | 18    | 431                        |
| acetaldehyde | 0.1 - 3                           | 1.53              | 0.8               | 8     | 36                         |

**Table 8.** Autonomous Results for Validation Testing Using the MU2 Challenge Cocktail. The large quantification error for benzene was ascribed to a co-elution with the 2-propanol. Note that (o-)xylene was present at levels below the VCAM requirement range and as such the quantification error was not included in the validation success criteria.

| Compound         | JPL<br>Requirement<br>Range<br>(ppm) | JSC Mean<br>(ppm) | JPL Mean<br>(ppm) | % Diff    |
|------------------|--------------------------------------|-------------------|-------------------|-----------|
| 2-propanol       | 1 - 10                               | 0.06              | 0.06              | -7        |
| dichloromethane  | 0.03 - 5                             | 0.06              | 0.05              | -19       |
| 2-butanone       | 0.5 - 5                              | 0.09              | 0.14              | 56        |
| ethyl acetate    | 1 - 10                               | 0.08              | 0.01              | -87       |
| 1-butanol        | 0.5 - 5                              | 0.00              | 0.02              | undefined |
| toluene          | 1 - 10                               | 0.02              | 0.00              | -76       |
| hexanal          | 0.1 - 2                              | 0.02              | 0.19              | 844       |
| (o-)xylene       | 1 - 10                               | 0.04              | 0.39              | 873       |
| OMCTS            | 0.05 - 1                             | 0.14              | 0.06              | -57       |
| acetone          | 0.5 - 5                              | 0.22              | 0.26              | 20        |
| acetaldehyde     | 0.1 - 3                              | 0.55              | 0.26              | -52       |
| ethanol          | 1 - 10                               | 0.7               | 0.23              | -69       |
| perfluoropropane | 10 - 100                             | 16                | 2.8               | -83       |

**Table 9. Autonomous Results for Validation Testing Using the MU3 Challenge Cocktail.** Note that hexanal and (o-)xylene were present at levels below the VCAM requirement range and as such the quantification error was not included in the validation success criteria.

Shown in Table 10 are the seven cocktails that were used for VCAM MCA validation. Mixes 1 and 3-7 were commercially procured from AirGas Products Inc. and certified for composition and concentration by mass spectral analysis. Mix 2 was terrestrial laboratory air. During pre-validation testing using Mixes 3-7 a calibrated instrument response curves for N<sub>2</sub>, O<sub>2</sub>, CO<sub>2</sub>, and Ar was generated that was then uploaded into VCAM for use by its autonomous measurement algorithms. The success criterion for MCA validation required that the percent relative standard deviation (RSD) for the derived concentration of each compound in the mixture be 20% or less for 3 MCA runs on Mixes 1 and 2. Summarized in Table 11 are the results of the validation testing showing VCAM had successfully passed the measurement criterion.

| Mix Designation      | % N2 | % O2 | % CO2 | % Ar |
|----------------------|------|------|-------|------|
| Mix 1 - ISS Simulant | 79   | 20   | 1     | 0    |
| Mix 2 - Room Air     | 78.1 | 20.9 | 0.036 | 0.93 |
| Mix 3 – Calibrant    | 72   | 26   | 1.5   | 0.5  |
| Mix 4 – Calibrant    | 75   | 24   | 0.8   | 0.2  |
| Mix 5 – Calibrant    | 76.4 | 23   | 0.6   | 0    |
| Mix 6 – Calibrant    | 78.7 | 21   | 0.3   | 0    |
| Mix 7 - Calibrant    | 82   | 18   | 0     | 0    |

**Table 10. Test and Calibration MCA Mixes Used in Verification Testing on the PFU.** An instrument response curve generated using Mixes 3-7 was generated during pre-validation testing. MCA verification success criteria were judged against instrument analysis of Mixes 1 and 2.

| Constituent     | Mix Name        | Actual % | Measured<br>Mean % | Std. Dev. % | % Diff. |
|-----------------|-----------------|----------|--------------------|-------------|---------|
| N <sub>2</sub>  | MCA-1 (ISS air) | 78.99    | 78.6               | 0.1         | 0.5     |
| O2              | MCA-1 (ISS air) | 20.00    | 20.4               | 0.1         | 2.0     |
| CO <sub>2</sub> | MCA-1 (ISS air) | 1.01     | 0.95               | 0.01        | 5.5     |
| Ar              | MCA-1 (ISS air) | 0.00     | 0.00               | trace       | trace   |
|                 |                 |          |                    |             |         |
| N2              | MCA-2 (lab air) | 78.1     | 78.0               | 0.3         | 0.1     |
| O2              | MCA-2 (lab air) | 20.9     | 21.1               | 0.4         | 1.0     |
| CO <sub>2</sub> | MCA-2 (lab air) | 0.038    | 0.04               | 0.02        | 5.3     |
| Ar              | MCA-2 (lab air) | 0.93     | 0.92               | 0.02        | 1.1     |

**Table 11. Autonomous Test Results for Validation of the MCA Measurement Mode.** Three repetitions of the measurement yielded %RSD for N<sub>2</sub>, O<sub>2</sub>, and CO<sub>2</sub> of 0.19%, 0.26%, and 5.4%, respectively.

# IV. Conclusion

Testing has demonstrated that VCAM has satisfied the performance criterion for compound identification, quantification, and precision. VCAM performed well even when analyzing species that appeared at concentrations below the required specification range. Terrestrial work involving the DU may provide some opportunities for improving VCAM performance at this low end of the instrument design specification range and extrapolating below that range. A highly detailed in-flight validation strategy and approach is imperative to realizing a flight demonstration success. When VCAM is used on-orbit, the only verified in-flight reference standard for VCAM will be the ISS Major Constituent Analyzer for N<sub>2</sub>, O<sub>2</sub>, and CO<sub>2</sub>. Unfortunately there will be no instrument available inflight for trace species verification. As such, a robust program involving co-temporal and co-spatial GSC sampling with VCAM measurements must be part of the in-flight validation approach.

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